

NEW UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity)

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Docket No. 94-36-3-US-D1

Total Pages in this Submission

TO THE ASSISTANT COMMISSIONER FOR PATENTS

Box Patent Application

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Enc	Enclosed are: Application Flements						
				Application Elements			
	1. X Filing fee as calculated and transmitted as described below						
2	2. Specification having pages and including the following:						
		a.	×	Descriptive Title of the Invention			
		b.	×	Cross References to Related Applications (if applicable)			
		C.		Statement Regarding Federally-sponsored Research/Development (if applicable)			
•		d.		Reference to Microfiche Appendix (if applicable)			
İ	e. 🛛 Background of the Invention						
	f. 🛛 Brief Summary of the Invention						
	g. 🛮 Brief Description of the Drawings (if drawings filed)						
	h. 🛛 Detailed Description						
		i.	×	Claim(s) as Classified Below			
		j.	×	Abstract of the Disclosure			
	3.	×	Dra	wing(s) (when necessary as prescribed by 35 USC 113)			
		a.	×	Formal			
		b.		Informal			
				Number of Sheets 1			

NEW UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity)

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Docket No. 94-36-3-US-D1

Total Pages in this Submission 44

Application Elements (Continued)

4.	X	Oath or Declaration					
	a.	☐ Newly executed (original or copy) ☐ Unexecuted					
	b.	Copy from a prior application (37 CFR 1.63(d)) (for continuation/divisional application only))				
	C.	☐ With Power of Attorney ☐ Without Power of Attorney					
5.	×	Incorporation By Reference (usable if Box 4b is checked) The entire disclosure of the prior application, from which a copy of the oath or declaration is Box 4b, is considered as being part of the disclosure of the accompanying application incorporated by reference therein.	s supplied under n and is hereby				
6.		Computer Program in Microfiche (Appendix)					
7.		Nucleotide and/or Amino Acid Sequence Submission (if applicable, all must be included)					
	a.	☐ Paper Copy					
	b.	☐ Computer Readable Copy (identical to computer copy)					
	C.	☐ Statement Verifying Identical Paper and Computer Readable Copy					
	A A It - Attend Donto						
		Accompanying Application Parts					
, 8.	×	Assignment Papers (cover sheet & document(s))					
9.		37 CFR 3.73(B) Statement (when there is an assignee)					
10.		English Translation Document (if applicable)					
11.	×	Information Disclosure Statement/PTO-1449 Copies of IDS Citations					
12.	×	Preliminary Amendment					
13.	×	Acknowledgment postcard					
14.	×	Certificate of Mailing					
		☐ First Class ☒ Express Mail (Specify Label No.): EI960111025US					
15.		Certified Copy of Priority Document(s) (if foreign priority is claimed)					

NEW UTILITY PATENT APPLICATION TRANSMITTAL (Large Entity)

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Total Pages in this Submission 44

P01ULRG/REV04

Accompanying Application Parts (Continued)

16. Addition	nal Enclosures (p	lease identity belo	ow): 					
		Fee Calcul	ation and Tr	ansmitta	al			
		CLAIMS	AS FILED			11		
For	#Filed	#Allowed	#Extra		Rate	Fee		
otal Claims	22	- 20 =	2	×	\$22.00	\$44.00		
ndep. Claims	2	- 3 =	0	x	\$82.00	\$0.00		
lultiple Depende	nt Claims (check	(if applicable)				\$0.00		
					BASIC FEE	\$790.00		
OTHER FEE (spe	ecify purpose)					\$0.00		
TOTAL FILING FEE \$834.00								
A check in the amount of to cover the filing fee is enclosed. M. The Commissioner is hereby authorized to charge and credit Deposit Account No. 06-2241								
as described below. A duplicate copy of this sheet is enclosed.								
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Page 3 of 3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Werenicz et al. Examiner: Unknown

Serial #: Unknown Group Art Unit: 3308

Filed: August 29, 1996 Docket: 94-36-3-US-D1

Title: METHOD FOR PRODUCING A CONTINUOUS THERMOPLASTIC

COATING & ARTICLES CONSTRUCTED THEREFROM

Honorable Commissioner of Patent and Trademarks Washington D.C. 20231

PRELIMINARY AMENDMENT

In the Specification

On p. 1, line 17, please insert after "This application is a" --divisional of application serial no. 08/705,578 which is a—

On p. 14, lines 3-9 replace:

"Lotryl" with --LOTRYL--

"Regalite" with --REGALITE--

"Petrobras" with --PETROBRAS--

"Catenex" with --CATENEX--

"Irganox" with --IRGANOX--

On p. 14, lines 3-9 replace:

"Escorene" with --ESCORENE--

"Regalite" with --REGALITE--

"Petrobras" with --PETROBRAS--

"Catenex" with --CATENEX--

"Irganox" with --IRGANOX--

EXPRESS MAIL NO E1960111025US On p. 18, at Example 14 insert --- after "Vestoplast"
On p. 19, at Examples 18, 19, 24-26 insert -- ®-- after "Lunatack"

In the Claims

Please cancel claims 13-32, add claims 33-42 and amend claims 1-2 as follows:

- A method of coating a thermoplastic composition [from a coating device] onto a substrate, said method comprising the steps of:
 - a) making [said] a thermoplastic composition flowable;
 - b) advancing [said] a substrate along a path;
 - c) dispensing a continuous film of said thermoplastic composition from [said] a coating device at a coating temperature wherein the complex viscosity of the thermoplastic composition is less than about 500 poise at about 1000 radians/second and ranges from about 100 poise to about 1,000 poise at about 1 radian/second;
 - <u>d</u>) suspending said film [of said composition being dispensed] between said coating device and said substrate [prior to];
 - e) contacting said film with said advancing substrate.
- 2. The method according to claim 1, wherein said substrate is [a] selected from the group consisting of textile material, heat sensitive materials, paper, hook and loop fastening webs, polyethylene materials, and nonwoven.

New Claims

- 33. A method of coating a hot melt adhesive onto a substrate, said method comprising the steps of:
 - a) making a hot melt adhesive composition flowable;

- b) advancing a substrate along a path;
- c) dispensing a continuous film of said hot melt adhesive composition from a coating device;
- d) suspending said film between said coating device and said substrate;
- e) contacting said film with said advancing substrate.
- 34. The method according to claim 33, wherein said substrate is selected from the group consisting of textile material, heat sensitive materials, paper, hook and loop fastening webs, polyethylene materials, and nonwoven.
- 35. The method according to claim 33, wherein the coating device is spaced from the path of the substrate at a distance between about 0.5 to about 20 mm.
- 36. The method according to claim 33, wherein the coating device is a slot nozzle.
- 37. The method according to claim 33, wherein the hot melt adhesive is dispensed onto the substrate such that the coating weight is less than about 30 g/m^2 .
- 38. The method according to claim 33, wherein the hot melt adhesive is coated at a rate of at least about 200 meters/minute.
- 39. The method according to claim 33, wherein the hot melt adhesive is released from the coating device at a temperature less than about 160°C.
- 40. The method according to claim 33, wherein the hot melt adhesive is released from the coating device at a temperature less than about 125°C.

- 41. The method according to claim 33, wherein the hot melt adhesive is released from the coating device at a temperature less than about 110°C.
- 42. The method according to claim 1, wherein the complex viscosity of the hot melt adhesive at the coating temperature is less than about 500 poise at about 1,000 radian/second and ranges form about 100 poise to about 1,000 poise at about 1 radian/second.

REMARKS

The amendments requested to the specification are being made to correctly claim priority in view of filing this divisional as well as for the purpose of insuring that the proprietary nature of trademarks is respected, as suggested by the Examiner in the parent case.

Claim 2 has been amended to recite substrates in addition to a textile material. Support for this amendment can be found on p. 4, lines 5-6; p. 5, line 11 and p. 8, line 4 and 7-8. Claim 13 has been incorporated into claim 1. An early allowance is respectfully requested.

Respectfully submitted,

4-8-90

Date

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METHOD FOR PRODUCING A CONTINUOUS THERMOPLASTIC COATING

AND ARTICLES CONSTRUCTED THEREFROM

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References to Related Applications

This application is a continuation-in-part of application Serial No. PCT/EP96/00377, filed January 30, 1996 which claims priority from PCT/EP95/00665, filed February 23, 1995, abandoned.

Field of the Invention

This invention relates to a non-contact coating method for producing a continuous coating and articles constructed therefrom. This invention further relates to a method for producing a textile material with a moisture-impermeable barrier layer and to a method for producing a moisture-absorbing article of hygiene which has such a barrier layer. This invention particularly relates to a textile material and hygienic disposable articles comprising a body fluid impermeable barrier layer which can be produced from said coating method. Preferably, the thermoplastic composition used in the coating method for producing the barrier layer exhibits certain rheological characteristics.

Background of the Invention

For various applications, materials are required which are impermeable to liquids such as water and body fluids, but at the same time have a textile character which is as close as possible to materials without the impermeability characteristics. One example of such application is hygienic articles such as disposable diapers, feminine napkins, panty liners, surgical drapes, bed pads, and the like. Such hygienic

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articles often have a substantial absorption capacity. To ensure that the liquid which is to be absorbed does not reach articles of clothing, such hygienic articles customarily have a continuous layer of a body fluid impermeable film on their garment facing side. Since touching this film material is an unwelcome sensation for a number of users, it has been suggested to cover the outward side of the film with a thin layer of textile material so that the article has a textile feel also on its exterior surface.

EP-A-O 187 728 teaches a disposable diaper with a barrier layer in the form of a plastics material film, typically consisting of a polyolefin, a polyacrylate, of PVC, nylon or other thermoplastic material. The barrier film is laminated, adhered or welded onto a thin nonwoven layer, which makes it necessary to prepare the film laminate off-line. As an alternative, it is suggested that the plastics material of the film is to be extrusion coated onto the nonwoven. The laminate, thus formed, is then used as the outermost material layer in such a way that the film layer is on the inside, so that the outer nonwoven layer provides the desired exterior texture. This production method is rather expensive. High molecular weight plastic materials such as polyethylene, polypropylene, polyacrylate and so on, have low melt flow indices and can (if at all) only be processed into impermeable films on very sophisticated machinery. This would also be true for the suggested direct extrusion coating which does not appear to have been reduced to practice yet. Separately producing the film, with subsequent laminating onto the nonwoven, is even more inefficacious in view of the additional production step required.

US patents 4,692,161 and 4,627,847 teach a leakage waste barrier for the edge of an absorbent hygienic article provided by coating a hot melt adhesive onto the edge area of a nonwoven sheet material. Depending on the actual type of application, this hot melt adhesive can also serve a constructive function, in combination with its function as a barrier, in that it can adhere the nonwoven to other materials of the hygienic article. The hot melt is to be coated in a conventional manner by slot nozzle coating, transfer coating, spray coating or other such methods. The above-mentioned US patents indicate that the hot melt coating must have a minimum thickness of 25 µm, preferably at least 75 µm, so that a continuous closed barrier layer is achieved.

Conventional slot nozzle coatings on uneven substrates such as nonwovens are

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typically done by keeping the slot nozzle in permanent contact with the substrate such that the nozzle lies on the substrate during the coating. It is unproblematic to coat hot melt adhesives onto uneven substrates with slot nozzles or spray coating methods, provided that only a discontinuous coating is required such as for constructive applications wherein the coating weight of the hot melt ranges from about 3 g/m^2 to about 10 g/m^2 . If, however, a continuous layer is to be created, this can only be done using these customary coating methods if the coating weight of the hot melt is greater than about 30 g/m^2 .

Such high coating weights are expensive. Furthermore, direct coating with a slot nozzle provides substantial mechanical and thermal stresses on the coated substrates, especially since the slot nozzle is heated during coating. Therefore, very sensitive substrates such as nonwovens made of very fine or low melting point fibers can not always be coated with hotmelt from a slot nozzle in a customary manner without damaging the substrate. Such problems can not be overcome when coating with heated coating rollers or spray coating with heated airstreams. The high coating weights of this prior art lead to increased stiffness of the coated substrate, thus impairing the textile character.

Similar problems occur in the production of hygienic articles and in other areas, such as fabric production, wherein the resulting materials are required to exhibit liquid impermeability, especially body fluid impermeability, with textile character which is as unimpaired as possible. This is especially pertinent for improving the comfort of the user. Presently, in such technical fields, production methods utilizing preformed laminated films are preferred. Therefore, there remains a need for a non-contact method capable of producing a continuous coating layer having low coating weights.

Summary of the Invention

The applicants have found a coating method that overcomes the aforementioned problems. The coating method employs a noncontact application wherein a thermoplastic composition is thermally made flowable and released from a coating device onto a substrate. The thermoplastic composition is thus coated onto the substrate without contact between said coating device and said substrate. Preferably,

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a liquid-impermeable, especially a body fluid impermeable, barrier wherein the textile character is not substantially impaired is produced. Since the method employs low coating weights of the thermoplastic composition, it eliminates the economic disadvantages of current methods as well as improves the tactile quality of the resulting article. Additionally, the method is suitable for coating a variety of heat sensitive materials. Preferably, the substrate is a "textile material" which in the context of this invention means not only a woven material made of yarn, but also any material made from fibers such as nonwoven, as well as nonwoven composites and the like, which materials play a major role in the area of hygienic article production. Since the coating device and substrate do not contact each other, the mechanical stresses on the substrate are much less than prior art methods.

For heat sensitive substrate, the thermoplastic composition is preferably coated at temperatures of less than about 160°C, more preferably less than about 125°C, and most preferably less than 110°C, to reduce the heat-induced stresses on the substrates being coated. This is advantageous for coating and mutually bonding thermally sensitive substrates.

The thermoplastic composition preferably exhibits certain rheological characteristics such that the complex viscosity at high shear rates (1,000 rad/sec) is less than about 500 poise and the complex viscosity at low shear rates (1 rad/sec) is between about 100 and about 1,000 poise. Some neat thermoplastic resins, such as typical film grade polyolefins, may be suitable for the method of the present invention. However, compounded hot melt adhesives are preferred due to the ability to independently control the visco-elastic properties, open time, etc. Compounded hot melts are advantageous to insure adequate adhesion to the carrier substrate or for delayed detackification of the coating after adherence to the substrate.

The resulting coating produced from said method is useful for a variety of applications wherein a consistent continuous coating is desired. Coating weights of less than 30 g/m² of the thermoplastic composition are preferred to reduce expenditure and improve the tactile quality of disposable hygienic articles. However, coating weights higher than 30 g/m² may be useful for other applications wherein reducing the mechanical and heat-induced stresses is of primary importance.

The resulting coating is preferable for producing a body fluid impermeable

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barrier layer in a disposable hygienic article having improved exterior tactile quality. The coating method is particularly advantageous for manufacturing as it employs fewer production steps than prior art coating methods. Improving productivity as well as reducing the coating weight mass per area results in coatings and corresponding articles that are less expensive than prior art.

The disposable article comprises at least one permeable substrate layer and at least one fluid impermeable barrier layer substantially adhered to the permeable substrate layer on at least one face, wherein the barrier layer comprises a thermoplastic compositions coated as a continuous film at an area weight of less than 30 g/m². Preferably, the barrier layer is produced from the coating method described herein. The permeable substrate is preferably a nonwoven web. However, paper, durable fabric, as well as any other material available as a roll good may be coated with this coating method. The permeable substrate may contribute significantly to the overall strength and integrity of the lamination. Preferably, the substrate exhibits sufficient strength such that it can not be torn easily by hand in either machine direction or in cross-direction.

This coating method is particularly useful for coating certain thermoplastic compositions that contribute desirable properties, yet are typically unsuitable for traditional extrusion die coating methods and can not be converted into a finished film.

Brief Description of the Drawing

Figure 1 depicts the method of the present invention wherein a continuous thermoplastic coating is formed and adhered to a carrier substrate.

Detailed Description of the Drawing

Substrate 1 (1) is advanced by the drive rolls (6) past a series of idle rollers (2) to ensure the web is in proper alignment prior to approaching the coating device (3). The coating device is located at a distance most often ranging from about 0.5 mm to about 20 mm, depending on the properties of the thermoplastic composition being coated. Substrate 2 (4) is optionally adhered to the coated surface of Substrate 1 by means of a nip roll (5). When adherence to a second substrate is intended, it is

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often preferred to position the nip roll closer to the coating device at a distance of about 25 cm from the coating device. In the preferred embodiment, Substrate 1 is a nonwoven web and Substrate 2 may be any material present in a hygienic article such as an absorbent, elastomeric strands or webs, tissue, films, coverstock materials such as nonwoven or perforated polyethylene, as well as any material that is not necessarily in the form of a web such as superabsorbent polymer.

Detailed Description of the Invention

In the method of the present invention, a melted hotmelt adhesive, preferably substantially air-free, is released from a coating or release device in such a way that it exits the device as a continuous film. A typical example for this is a slot nozzle, as it has previously been used for coating in direct contact with a substrate. Thus, melt coating devices which are already at hand can be reset for use according to the invention in that the slot nozzle is lifted off the substrate and is adjusted to have a suitable distance from the substrate.

When the viscous but flowable molten adhesive leaves the coating device, it does not contact the substrate immediately, but rather travels for a distance as a continuous coating film suspended above the substrate without touching either the device or the substrate. The distance between the coating device and the substrate ranges from about 0.5 mm to about 20 mm. It is possible that at suitable machine speed settings, and with specific adhesives or other coating materials, the distance can be even greater than 20 mm. The distance is largely dictated by the viscosity, flow properties such as shear thinning behavior, line speed and open time of the thermoplastic composition being coated. It is surmised that the thermoplastic composition builds sufficiently in viscosity and cohesive strength to the extent that any filaments or fibers present on the substrate surface cannot penetrate the coating, yet the thermoplastic composition is molten or soft enough to adequately adhere to the substrate.

It has been shown to be especially advantageous, that the coating later contacts the substrate in a substantially horizontal direction rather than in a vertical direction.

To realize this advantage, a roller can be provided in the path of movement of the substrate to give the substrate a substantially vertical, upward direction, as the

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substrate passes the coating device. Additionally, the coating device, such as a slot nozzle, can be provided substantially horizontally beside the roller so that the coating travels from the side towards the surface of the substrate.

The diameter of the coating role is preferably about 15 mm to about 50 mm in diameter with the nozzle above the center of the coating roll such that the angle at which the thermoplastic coating contacts the substrate is less than about 60° as the substrate is moving away from the nozzle. The coating head is adjusted by one of ordinary skill in the art to optimize for even flow and distribution of the thermoplastic coating over the entire width of the application.

Thereafter, the sufficiently viscous coating contacts the substrate surface and adheres to the surface without deeply penetrating into the substrate. Particularly for coating thermoplastic compositions that are relatively tack free upon cooling, there is a distinct interface between the coating and the substrate which is evident by the ability to remove the coating as a continuous film from the substrate. This interface is also present when a tacky thermoplastic composition is employed. However, in this instance since the coating and the substrate are inseparable, the interface is not quite so evident. When the substrate is preferably a textile material such as a nonwoven, the thus produced material comprises the textile substrate layer and a coating, preferably a hotmelt barrier layer. If the thermoplastic coating is of such a composition that it substantially detackifies after sufficient cooling, the laminate of the coated substrate, thus formed, can be rolled up and stored. The laminate can then be used at some later time e.g. as a body fluid impermeable backsheet having improved tactile quality in a disposable hygienic article. The laminate can be bonded by any suitable bonding technique including ultrasonic bonding, heat welding, or more commonly adhesive bonding.

Preferably, the coating of the barrier layer is done "inline" immediately before any further processing of the thus produced coated textile laminate. In such a case, the surface of the barrier layer which is pointing away from the substrate and is still sufficiently tacky can be used for a constructive adhesion step and therefore can also serve to bond the coated textile material to other elements of a hygienic article. Other elements that could be simultaneously bonded in this manner during the formation of the barrier layer include absorbent, elastomeric strands or webs, tissue, films,

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coverstock materials such as nonwoven or perforated films, as well as any other material that is not necessarily in the form of a web such as superabsorbent polymer. This method has been found to be particularly advantageous for bonding porous web including hook and loop fastening webs such as Velcro.

Since the hot melt coating can be provided at extremely low temperatures, materials can also be provided with barrier layers which are too sensitive mechanically and/or thermally for customary coating methods. Such sensitive materials include low gauge polyethylene materials, low basis weight nonwovens and the like.

A substantial advantage of the invention is that continuous, sufficiently impermeable barrier layers can be made from hotmelts at very low coating weights. Even with customary commercially available hotmelts, closed barrier layers can be produced at coating weight of not more than 30 g/m^2 , and generally, it is easily possible to achieve coating weights between 10 g/m^2 and 20 g/m^2 and most preferably less than 10 g/m^2 . As previously stated, the prior art coating of hot melts according to customary methods for forming edge leakage barriers, as in US patent 4,692,161, requires area weights of about 70 g/m^2 to create the preferred film thickness of around $75 \mu\text{m}$. At thickness of $25 \mu\text{m}$, the suggested minimum according to this art, the contact-coated layer is perforated by substrate fibre, and is not closed.

The very thin barrier layers which can be produced according to the invention do not only contribute to the economical advantages of the inventive method, but also make it possible to achieve a very much reduced stiffness of the material, which thus comes much closer, in its properties, to a textile material which is not provided with a barrier layer at all. The reduced stiffness is achieved not only by the reduced coating weight thickness but also due to the ability to coat thermoplastic materials that are typically unsuitable for traditional extrusion die coating methods and can not be converted into a finished film. The film itself, formed from this method typically does not exhibit sufficient strength unless it is supported by the substrate upon which it is coated.

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The Thermoplastic Composition

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polyolefins, especially polyethylene, polypropylene, amorphous polyolefins such as Vestoplast 703® (Hūls), metallocene polyolefins, and the like, may be suitable thermoplastic materials for the coating method of the present invention. However, hot melt adhesives are preferred due to the ability to independently tailor the viscoelastic properties, open time, tack, and various other properties. Hot melt adhesives commonly have viscosity profiles that allow for processing at very low temperatures. Typical hotmelts are fluid enough for such processing at temperatures ranging from about 60°C to 110°C.

More preferably, the thermoplastic composition exhibits certain rheological characteristics such that a continuous, body fluid impermeable coating can be produced at coating weights of less than about 30 g/m². In general, the rheological properties preferably fall within a rheological window wherein the complex viscosity at high shear rates (1,000 rad/sec) is less than about 500 poise and the complex viscosity at low shear rates (< 1 rad/sec) is between about 100 and about 1,000 poise. In other words, preferable thermoplastic compositions exhibit Newtonian regions at low shear rates and shear thinning at higher shear rates. Thermoplastic compositions having wide windows of application are those in which the composition exhibits the appropriate rheological properties at a variety of application settings, particularly low temperatures. Narrow application windows are those in which the rheological parameters are only met under very specific conditions. Amorphous polyolefins based hot melt adhesives such as Lunatack® D-8370 (H.B. Fuller Company) tend to exhibit relatively wide application windows whereas block copolymer based hot melt adhesives tend to exhibit narrow application windows.

Data generated that supports this rheological window is depicted in Table I. The test procedures used to determine the rheological data are described in detail hereinafter. The applicants surmise that the high shear rate data relates to the processing conditions at the slot die exit. A composition with too high of a complex viscosity at 1,000 radians/sec would require excessive pump pressure to exit the coating device. A die with a shim gap larger than 3 mm could be used to process these materials but a higher coating weight may result.

The low shear rate data relates to the settling of the coating on the substrate during the time it is suspended above the substrate. If the low shear value is too

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high, the coating may not adhere adequately to the substrate and/or the thermoplastic composition builds up at the nozzle causing a streaked, discontinuous coating. If the low shear viscosity is too low, the coating may seep into the substrate, causing poor barrier properties.

Extensional viscosity, which was not measured can also strongly influence the melt strength. Higher levels of branching or the addition of a small concentration of a high molecular weight material can strongly influence the melt strength. More preferred are compositions that meet the target rheological parameters at low application temperatures of less than about 160°C, more preferably less than about 125°C and most preferably less than about 110°C.

Accordingly, many known hot melt adhesive compositions are well suited for use in the coating method of this invention. Hot melt adhesives typically comprise at least one thermoplastic polymer, at least one plasticizer and at least one tackifying resin. Preferably, such suitable hotmelts comprise up to 40% by weight of thermoplastic polymer, up to 40% by weight of a plasticizer and up to 70% by weight of tackifying resin.

With respect to the thermoplastic polymer, atactic polyalphaolefins such as Vestoplast® 708 (Hüls) and synthetic rubbers such as S-EB-S block copolymers have been found to be especially suited, particularly those as used in hot melt adhesives such as Lunatack® D-3964 (H.B. Fuller). Further, however, other thermoplastic polymers are suitable, such as ethylenic copolymers including ethylene-vinyl acetate, ethylene-methyl acrylate, copolymers or other synthetic rubbers as available in commerce under the tradenames Kraton®, Solprene®, and Stereon®.

In the case of polyolefins and ethylenic copolymers, polymer concentrations as high as 100% may be suitable. It should be noted that compositions useful for traditional extrusion die coating are typically not suitable uncompounded for the coating method described herein. Such commercially available neat resins such as polypropylene and polyethylene do not have a sufficiently low enough complex viscosity at low temperatures, preferably less than 160°C, to be coated in this manner. Furthermore, a single unimodal polymer of sufficiently low enough complex viscosity is typically high in density, greater than about .90 g/cm². Due to being highly crystalline in nature, such neat polymers do not possess the proper balance of

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visco-elastic properties to produce fluid-impermeable coatings at low coating weights.

Plasticizers and tackifying resins used in hot melt adhesives are known. Oils such as naphthenic oils are preferred plasticizers. As for tackifying resins, those resins already known for such purposes are generally suitable, especially hydrocarbon resins, ester resins and other such compatible resins. The components are mixed and processed in a known manner to prepare the hotmelts which can be used according to this invention.

With suitable hot melts, such as those described in DE-A-41 21 716, it is also possible to make materials which are impermeable to liquid water, yet water vapor permeable rendering the coating "breathable".

In addition to commonly known hot melt adhesives, thermoplastic compositions comprising a water soluble, saline (body fluid) insoluble copolyester such as Eastman AQ 1350[®], commercially available from Eastman, are also particularly useful for creating barrier films that are impervious to body fluid, yet readily water soluble. This feature is of particular interest for creating flushable and compostable disposable hygienic products. Furthermore, there may be applications wherein water permeability is desired. Accordingly, this coating method may also be suitable for coating water permeable, water soluble and/or biodegradable thermoplastic materials.

Hereinafter, the invention will be further depicted by the following nonlimiting examples.

Embodiment Example 1:

Several hot melts which slightly differ from each other in composition were formulated in the following composition ranges:

- 20 25% naphthenic oil
- 30 40% atactic polyolefin(s)
- 35 45% hydrocarbon resin

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Embodiment Example 2:

Several hot melts were formulated within the following range limits:

- 15 20% SIS-block copolymer
- 15 25% naphthenic oil
- 5 50 65% ester resin

Embodiment Example 3:

As a commercially available hot melt adhesive, the "Lunatack D 8370" product was used, which is available from H. B. Fuller GmbH. This is a hot melt adhesive comprising about 35% polyolefin, about 40% hydrocarbon resin with a cyclopentadiene component, about 15% polyisobutylene and about 10% naphthenic oil.

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Examples 4-28:

Table 1 depicts rheological data on examples 4 through 16 and examples 18 through 28. Column 2 of Table 1 depicts the reference temperature for the rheological parameters as well as the coating application temperature for each sample. Table 2 and Table 3 depict the chemical description of examples 4 through 28 as well as the coating parameters for those examples in which a continuous coating resulted. A consistent continuous coating was not able to be produced with Samples 4 through 9 at the temperature indicated in Column 2. The applicants surmise that the inability to produce a continuous coating is due to the complex viscosity being greater than about 1000 poise at about 1 rad/sec. By comparing examples 5 with 14 and 4 with 10, the complex viscosity at 1 rad/sec can be forced into the rheological window by increasing the temperature. By comparing example 7 with 16, the applicants have demonstrated the relatively narrow rheological window of Lunatack® D-3964. At 90°C D-3964 exhibits too high of a complex viscosity at 1 rad/sec. At 110°C, D-3964 exhibits too low of a complex viscosity at 1 rad/sec, causing the material to soak into the substrate. The applicants surmise a temperature exists between 90°C and -110°C wherein D-3964 would produce a continuous coating. However, a

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thermoplastic composition exhibiting such a narrow rheological window would have little chance of commercial success.

Example 14 exhibits the utility of blending a thermoplastic composition that does not meet the rheological window with another material such that the resulting composition is useful for producing a continuous coating. In this particular example, since D-3964 exhibits too low of a complex viscosity at 1 rad/sec to produce a continuous coating at a coat weight of about 10 g/m², it is blended with a material to raise the complex viscosity at 1 rad/sec and to improve the shear thinning properties such that the blend exhibits the preferable rheological properties. Alternatively, the low complex viscosity at 1 rad/sec is unproblematic for coating weights of about 20 g/m² and higher as illustrated by examples 24 through 26.

Examples exhibiting too high of a complex viscosity at 1 rad/sec, such as examples 4 through 9 can be blended with compatible materials to lower the complex viscosity such that the blended material may be coated at the preferable application temperature of less than 160°C.

The thermoplastic composition of examples 18 and 19 is particularly preferred since the resulting coating demonstrated no change in water penetration after being aged for 3 days at 60°C.

The compositions of experimental samples D573BD7 and D573BD8 are depicted below.

Experimental Sample D573BD7:

	Parts (pph)	<u>Tradename</u>	Chemical Description
	33.0	Lotryl 35 BA 40 (Elf Atochem)	Ethylene n-butyl acrylate copolymer
25	24.7	ECR-179 A (Exxon)	Tackifying resin
	20.0	Regalite R 125 (Hercules)	Tackifying resin
	15.0	Petrobras K 521 (A. Kochen, Gm	bH) Wax
	5.0	Catenex P941 (Shell)	Plasticizing oil
	.15	Irganox 1010 (Ciba Geigy)	Antioxidant
30	15	Irganox PS 800 (Ciba Geigy)	Antioxidant

Experimental Sample D573BD8:

	Parts (pph)	<u>Tradename</u>	Chemical Description
	33.0	Escorene UL 02528	Ethylene vinyl-acetate
	24.7	ECR-179 A (Exxon)	Tackifying resin
5	20.0	Regalite R 125 (Hercules)	Tackifying resin
	15.0	Petrobras K 521 (A. Kochen, Gm	bH) Wax
	5.0	Catenex P941 (Shell)	Plasticizing oil
	.15	Irganox 1010 (Ciba Geigy)	Antioxidant
	.15	Irganox PS 800 (Ciba Geigy)	Antioxidant

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Test Methods

Coating Procedure

The hot melts according to embodiment examples 1 through 3 were placed in a customary processing machine provided with a slot nozzle such as Nordson EP 51. The slot nozzle was provided horizontally facing a roller over which a 23 g/m² basis weight spunbond polypropylene nonwoven (Corovin® A23A40 Corosoft-Plus, Corovin GmgH) was led in an upward direction. The distance between the slot nozzle and the substrate was 2 mm, at a nozzle slot length of 70 mm. The web speed of the nonwoven was 25 m/min. At a system pressure of about 53 bar and a release temperature of the hot melt of approximately 100°C, the hot melt was coated onto the substrate, where it formed a closed barrier layer. Immediately thereafter, the thus coated substrate was adhered to a customary absorptive body (tissue). In each case, a reliable adhesive bond between substrate and tissue was provided, and in each case, the hot melt barrier layer formed between the tissue and the substrate was found to be completely liquid-impermeable. Processing was without any problems. The coating weight was an average of 21 g/m². At corresponding fine adjustment of release temperature of hot melt, system pressure, distance between slot nozzle and substrate, machine speed etc. etc., it was systematically possible to form water-tight closed barrier layers at area weights of less than 20 g/m² on this substrate.

Examples 4 through 28 were coated in a similar manner as examples 1 - 3 - with the exception that the coating was not adhered to tissue. The application

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conditions and rheological data of the adhesive compositions are depicted in Table 1. A system back pressure ranging from about 40 to about 65 bar was obtained during coating of examples 10 through 28.

5 Rheological Testing

The rheological data was generated from a dynamic mechanical spectrometer such as a Rheometric Scientific RDS 7700 (10,000 g/cm transducer, 10 g/cm -<10,000 g/cm torque). A master curve of G' (shear storage modulus), G" (shear loss modulus) and complex viscosity as a function of frequency was obtained through time temperature superposition. During testing the sample was loaded at the upper test temperature between 50 mm diameter parallel plate discs with a 1 to 2 mm gap. After allowing the sample temperature to stabilize for at least about 10 minutes, a frequency sweep was performed from about 0.1 to about 100 radians per second. Upon the completion of the frequency sweep, the sample temperature was lowered to the next temperature and the procedure repeated. The strain amplitude was adjusted to improve the resolution and ranged from about 20% to about 40%. After the frequency sweep was completed at the final, lowest temperature, time-temperature superposition was used to overlay the data into a single master curve at the application temperature. If the actual coating temperature was not one of the actual temperatures tested, the Williams, Landel, Ferry (WLF) (Ferry, J.D. Viscoelastic Properties of Polymers, 3rd Ed., Wiley: NY, 1980) equation was used to obtain the master curve.

Time-temperature superposition can be applied for amorphous compositions as well as for crystalline compositions for temperatures above the melt temperature.

Capillary rheometry could be used to measure the viscosity at high shear rates.

Water permeability

The coated nonwoven was tested according to EDANA 160.0-89"Wet

Barrier" test method. The samples were conditioned for 24 hours at 50% +/- 2%

relative humidity, 23°C +/- 2°C prior to testing. The samples were fixed (hot melt

surface facing the water) to a cylindrical vessel, fitted with a bolted ring for clamping

-samples with a circular test area. The water pressure was increased recording the

values the first drop (rather than the third as described in ERT 120.1-80) appeared on the surface of the nonwoven.

Table 1

Example	Temp.	Complex Viscosity	Complex Viacosity	G' 1 rad/sec	Crossover Frequency	Tan delta @ l rad/sec	Slope = Visc @	Continuous Coating Formed
	(00)	l rad/sec (poise)	10'rad/sec (poise)	(dynes/cm²)	(rad/sec)		rad/sec	yes/no
4	125	15000	100	10000	1	1	150	no
2	96	10000	300	3000	300	3	33	ou
9	120	4500	1500	300	1000	30	4.5	ou
7	06	3000	100	400	50	7	30	not tested
8	110	2000	500	700	10000	3	4	no
6	140	1000	500	50	>1000	70	2	no
10	160	200	200	5	>1000	4	1.25	уев
11	125	800	100	5000	20	1	8	уев
12	125	800	100	100	1000	10	8	уев
13	125	300	50	200	1	1	9	not tested
14	110	300	50	20	7000	20	9	уев
15	128	100	80	10	1000	10	1.25	уев
16 & 24-26	110	100	3.5	8	100	25	28	ou
18	130	350	06	40	>1000	10	4	уев
19	110	800	130	130	1000	8	9	уев
20	140	120	60	Ţ	>1000	50	2	yes
21	110	500	200	20	1000	20	2.5	уев
22 & 23	130	250	120	1.0	>1000	30	2	yes
৺	120	120	60	1	1100	100	2	уев

Table 2

Example	Tradename (s)	Chemical Description	Coating	Speed	Permeability
•			(GSM)	M/MIN	pressure
4	347-BD-19 (H.B. Fuller)	atactic polyolefin hotmelt adhesive (HMA)			
S	4 C1	SEBS block copolymer/hydrocarbon resin/napthenic oil HMA + atactic polyolefin			
9	Eastman AQ® 1350	water dispersible copolyester (WO 95/18191)			
7	D-3964	SEBS block copolymer/hydrocarbon resin/napthenic oil HMA			
80	NP-2085 (HBF)	urethane			
6	Eastman AQ® 1350	see Example 6			
10	Eastman AQ® 1350	see Example 6			
11	Vestoplast [®] 703 (Huls)	atactic polyolefin	22	30	ı
12	347-BD-33 (HBF)	atactic polyolefin HMA	10	30	100
13	Vestoplast® 703 + 10% Paraflint H4	atactic polyolefin + Fischer Tropsch wax			
14	D-3964 + 10% Vestoplast 750	SEBS block copolymer/hydrocarbon resin/napthenic oil HMA + atactic polyolefin	9-11	34	50
15	D-8370	atactic polyolefin HMA	12	30	. 94
16	D-3964	see Example 7	8	30	
17	Vestoplast® 750 + 10% Wax	see Example 13	15-16	30	•

Table 3

Example	Tradename(s)	Chemical Description	Coating	Speed	Permeability cm³ of H ₂ 0
		Coating Temperature	(GSM)	M/MIN	pressure
18	Lunatack D-9105	atactic polyolefin HMA	7-8	700	80
	(H.B. Fuller)	130°C			
19	Lunatack D-9105	atactic polyolefin HMA 110°C	10-11	50	230
20	Experimental Sample	ethylene n-butyl acrylate copolymer HMA	81	200	20
	D573BD7	140°C			
21	Experimental Sample	ethylene vinyl-actetate copolymer HMA	8-9	200	not tested
	D573BD8	110°C			
22	Experimental Sample	ethylene vinyl-acetate copolymer HMA	38	30	220
	D573BD8	130°C			
23	Experimental Sample	ethylene vinyl-acetate copolymer HMA	15-16	001	20
	D573BD8	130°C			
24	Linatack D-3964	see Example 7	62	30	300
	(H.B. Fuller)	110°C		ŕ	
25	Lunatack D-3964	see Example 7 110°C	27	30	20
26	Lunatack D-3964	see Example 7 110°C	20	150	not tested
27	Experimental Sample	SIS block copolymer HMA	27	100	20
	D181BD18ZP	120°C			
28	Experimental Sample	SIS Block Copolymer HMA	12	200	not tested
	D-181BD18ZP	120°C			

What is claimed is:

- 1. A method of coating a thermoplastic composition from a coating device onto a substrate, said method comprising the steps of:
 making said thermoplastic composition flowable;
 advancing said substrate along a path;
 dispensing a continuous film of said compositions from said coating device;
 suspending said film of said composition being dispensed between said coating device and said substrate prior to contacting said film with said advancing substrate.
- 2. The method according to claim 1, wherein said substrate is a textile material.
- 3. The method according to claim 1, wherein the coating device is spaced from the path of the substrate at a distance between about 0.5 to about 20 mm.
- 4. The method according to claim 3, wherein the distance between the coating device and the substrate is less than about 10 mm.
- 5. The method according to claim 1, wherein the coating device is a slot nozzle.
- 6. The method according to claim 5, wherein said slot nozzle has a shim gap of less than 5 mm.
- 7. The method according to claim 1, wherein the substrate is directed substantially vertically immediately after passing the coating device.
- 8. The method according to claim 1, wherein the thermoplastic composition is dispensed onto the substrate such that the coating weight is less than about 30 g/m².
- 9. The method according to claim 1, wherein the thermoplastic composition is coated at a rate of at least about 200 meters/min.

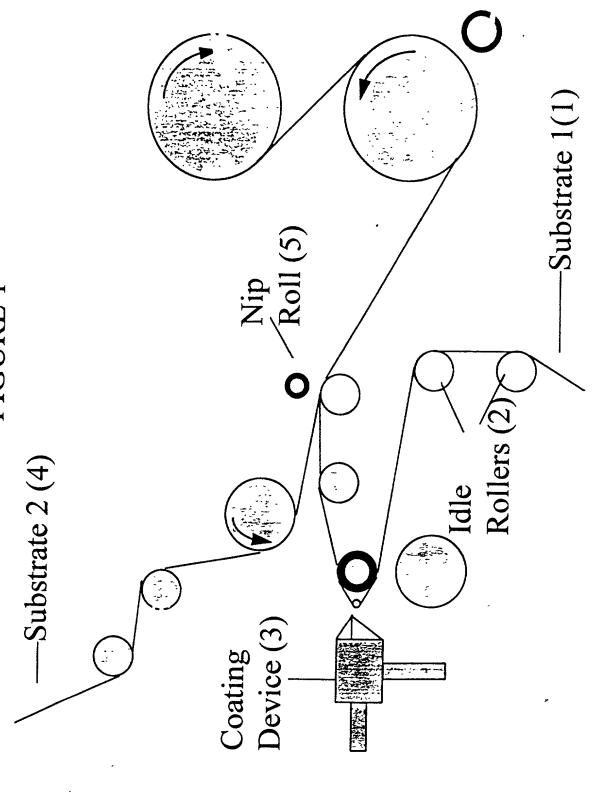
- 10. The method according to claim 1, wherein the thermoplastic composition is released from the coating device at a temperature of less than about 160°C.
- 11. The method according to claim 1, wherein the thermoplastic composition is released from the coating device at a temperature of less than about 125°C.
- 12. The method according to claim 1, wherein the thermoplastic composition is released from the coating device at a temperature of less than about 110°C.
- 13. The method according to claim 1, wherein the complex viscosity of the thermoplastic composition at the coating temperature is less than about 500 poise at about 1,000 radians/sec and ranges from about 100 to about 1,000 poise at about 1 radian/sec.
- 14. A disposable article comprising at least one permeable substrate layer and at least one fluid impermeable barrier layer substantially adhered to the permeable substrate layer on at least one face, wherein said barrier layer comprises a thermoplastic composition coated as a continuous film at an area weight of less than 30 g/m².
- 15. The disposable article of claim 14, wherein the article further comprises at least one absorbent material.
- 16. The disposable article of claim 14, wherein the barrier layer is moisture-vapor permeable.
- 17. The disposable article of claim 14, wherein the outer layer is a textile material.
- 18. The article of claim 14, wherein the barrier layer comprises a thermoplastic composition such that the complex viscosity of the thermoplastic composition at the coating temperature is less than about 500 poise at about 1,000 radians/sec.

- 19. The article of claim 18, wherein the barrier layer comprises at least one polymer selected from the group consisting of block copolymers, water dispersible copolyesters, ethylenic copolymers, polyolefins, metallocene polyolefins, atactic polyolefins and mixtures thereof.
- 20. The article of claim 18, wherein the coating temperature is less than about 160°C.
- 21. The article of claim 14, wherein the barrier layer comprises a thermoplastic composition such that the complex viscosity of the thermoplastic composition at the coating temperature ranges from about 100 to 1,000 poise at about 1 radian/sec.
- 22. The article of claim 21, wherein the barrier layer comprises at least one polymer selected from the group consisting of block copolymers, water dispersible copolyesters, ethylenic copolymers, polyolefins, metallocene polyolefins, atactic polyolefins and mixtures thereof.
- 23. The article of claim 21, wherein the coating temperature is less than about 160°C.
- 24. The article of claim 14, wherein the thermoplastic composition forming the barrier layer is subsequently bonded to at least one other material.
- 25. The article of claim 14, wherein the thermoplastic composition is bonded to at least one other material inline after coating of the thermoplastic composition.
- 26. The article of claim 14, wherein the barrier layer comprises a hot melt adhesive.
- 27. The article of claim 14, wherein the area weight of the thermoplatic composition is less than about 20 g/m².
- 28. The article of claim 14, wherein the area weight of the thermoplastic composition is less than about 10 g/m^2 .

- 29. An article comprising a body fluid impermeable barrier wherein the area weight of said barrier layer is less than about 20 g/m².
- 30. The article of claim 29, wherein the area weight of said barrier layer is less than about 10 g/m^2 .
- 31. A disposable article comprising at least one permeable substrate layer and at least one fluid impermeable barrier layer adhered to the permeable substrate layer on at least one face, wherein said barrier layer is a coating composition dispensed from a coating device as continuous film at a temperature of less than 160°C, and said coating device is spaced from the path of the substrate at a distance between about 0.5 to about 20 mm.
- 32. A thermoplastic coating comprising a thermoplastic composition wherein the complex viscosity at the coating temperature of said composition is less than about 500 poise at about 1,000 radians/sec and ranges from about 100 to about 1000 poise at 1 radian/sec, wherein said composition can be coated to produce a fluid impermeable barrier at an area weight of less than about 30 g/m².

Abstract

This invention relates to a non-contact coating method for producing a continuous coating and articles constructed therefrom. This invention further relates to a method for producing a textile material with a moisture-impermeable barrier layer and to a method for producing a moisture-absorbing article of hygiene which has such a barrier layer. This invention particularly relates to a textile material and hygienic disposable articles comprising a body fluid impermeable barrier layer produced from said coating method. Preferably, the thermoplastic composition used in the method for producing the barrier layer exhibits certain rheological characteristics.



H.B. FULLER COMPANY

United States Patent Application

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor I hereby declare that: my residence, post office address and citizenship are as stated below next to my name; that

I verily believe I am the original, first and sole inventor (if only one name is listed below) or a-joint inventor (if plural inventors are named below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD FOR PRODUCING CONTINUOUS THERMOPLASTIC COATING AND ARTICLES CONSTRUCTED THEREFROM

The specification of which a is attached hereto bX_ was filed on August 29, 19 PCT-filed application) described at reviewed and for which I solicit a limit I hereby state that I have reviewed amended by any amendment referring I acknowledge the duty to disclose	nd claimed in international no	_ filed and as amended on	(II any), winch I have including the claims, as				
37, Code of Federal Regulation, §	1.56 (see page 3 attached hereto).						
inventor's certificate listed below a filing date before that of the applaration and applications have b	I hereby claim foreign priority benefits under Title 35, United States code, § 119/365 of any foreign application(s) for patent of inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on the basis of which priority is claimed. a no such applications have been filed. bX_ such applications have been filed as follows:						
FOREIGN APPLICATION(S), IF ANY, CLAIMING PRIORITY UNDER 35 USC § 119							
COUNTRY	DATE OF ISSUE						
PCT	PCT/EP95/00665	23 February, 1995	Abandoned				
PCT	Donding						
ALL FOREIGN APPLICATION(S), IF ANY, FILED BEFORE THE PRIORITY APPLICATIONS(S)							
COUNTRY	APPLICATION NUMBER	DATE OF FILING (Day, Month, Year)	DATE OF ISSUE (Day, Month, Year)				

I hereby claim the benefit under Title 35, United States Code, § 120/365 of any United States and PCT international application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

NO. E1960111025US

U.S. APPLICATION NUMBER	DATE OF FILING (Day, Month, Year)	STATUS (Patented, Pending, Abandoned)
PCT/EP96/00377	30 January, 1996	Pending

I hereby appoint the following attorney(s) and/or patent agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith: Nancy N. Quan, Agent Reg. #36,248; Carolyn A. Fischer, Agent Reg. #39,091

I hereby authorize them to act and rely on instructions from and communicate directly with the person/assignee/attorney/firm/organization/who/which first sends/sent this case to them and by whom/which I hereby declare that I have consented after full disclosure to be represented unless/until I instruct H.B. Fuller to the contrary.

Please direct all correspondence in this case to H.B. Fuller at the address indicated below:

H.B. Fuller Company 3450 LaBore Road St. Paul, MN 55110

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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Signatur	e of Inventor 207	
Date	13/29/96	

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- A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with the Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is canceled or withdrawn from consideration, or the application becomes abandoned. Information material to the patentability of a claim that is canceled or withdrawn from consideration need not be submitted if the information is not material to the patentability of any claim remaining under consideration in the application. There is no duty to submit information which is not material to the patentability of any existing claim. The duty to disclose all information known to be material to patentability is deemed to be satisfied if all information known to be material to patentability of any claim issued in a patent was cited by the Office or submitted to the Office in the manner prescribed by §§ 1.97(b)-(d) and 1.98. However, no patent will be granted on an application in connection with which fraud-on the Office was practiced or attempted or the duty of disclosure was violated through bad faith or intentional misconduct. The Office encourages applicants to carefully examine:
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- Under this section, information is material to patentability when it is not cumulative to information already of
 - It establishes, by itself or in combination with other information, a prima facie case of unpatentability of a claim;
 - It refutes, or is inconsistent with, a position the applicant takes in:
 - Opposing an argument of unpatentability relied on by the Office, or
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(b) Under this section, information is may record or being made of record in the application, and

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